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» Energetic Materials

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Author: **Subject: Ammonium perchlorate without DIY anodes?**

greenlight
International Hazard
★★★★★

posted on 7-10-2015 at 07:06

Posts: 620
Registered: 3-11-2014
Member Is Offline

Mood: Energetic

It should be Hydrogen coming from the vent tube in the cell.
I lead mine into a sodium hydroxide solution but never noticed any real bubbling.
What temperature is your cell running at?

I ran my 900ml cell for 100 hours total at 5 volt 20 amp and didn't notice any chlorate precipitating on the bottom for the first day or so.
The chlorine cell is normal and was present every time i opened my cell too.

The only use for an atomic bomb is to keep somebody else from using one.
George Wald

PROFILE FIND

ecos
National Hazard
★★★★

posted on 7-10-2015 at 14:17

Posts: 464
Registered: 6-3-2014
Member Is Offline

Mood: Learning !

> **Quote:** Originally posted by greenlight
>
> It should be Hydrogen coming from the vent tube in the cell.
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> The chlorine cell is normal and was present every time i opened my cell too.

Thanks Greenlight for your answer, I really appreciate it.

I checked my cell and it is hot but not sure about the temperature. I think it is around 40-60 degrees Celsius.

I use a PC power supply. I used the 5v terminal but noticed that the volts drops till 3.5v when it is connected to the cell. the current is 4A.
The power supply is capable to produce 20A on 5v but not sure why it drops down to 3.5v.

what kind of power supply do you use?

two days of running and there is no sign of any KClO3 in the bottom.

[Edited on 7-10-2015 by ecos]

PROFILE FIND

greenlight
International Hazard
★★★★★

posted on 7-10-2015 at 18:28

Posts: 620
Registered: 3-11-2014
Member Is Offline

Mood: Energetic

I never had a meter to see what voltage or amperage was running through the cell but mine did get hot to about 60 or 70 degrees. I had to use a water bath at times because it would have gone higher.
I used this power supply:

http://www.ebay.com/itm/S-100-5-Super-Stable-Power-supply-un...

Have you been running the cell non-stop or have you let it cool down to see if an chlorate precipitates when the temperature drops back down?

The only use for an atomic bomb is to keep somebody else from using one.
George Wald

PROFILE FIND

hyfalcon
International Hazard
★★★★★

posted on 8-10-2015 at 02:23

Posts: 1003
Registered: 29-3-2012
Member Is Offline

Mood: No Mood

If you are using potassium salts in a concentrated solution to start, you won't have to cool the electrolyte down. Due to it's lower solubility, the potassium chlorate starts falling out of solution on it's own.

PROFILE FIND

greenlight
International Hazard
★★★★★

posted on 8-10-2015 at 03:00

Posts: 620
Registered: 3-11-2014
Member Is Offline

Mood: Energetic

Yes, my cell precipitated chlorate after the first day but when I cooled it down to room temp even more fell out of the solution.
Is it possible that Ecos does have chlorate but so little at the moment that it is staying dissolved while he is running the cell because of the temperature of the electrolyte.

The only use for an atomic bomb is to keep somebody else from using one.
George Wald

PROFILE FIND

ecos
National Hazard
★★★★

posted on 8-10-2015 at 03:02

Posts: 464
Registered: 6-3-2014
Member Is Offline

Mood: Learning !

I didnt stop the cell to check the chlorate.
I only had power outage for several hours.would this destroy the chlorate formation?

I cant see any participation in the bottom of the cell and it is running for 3 days.

PROFILE FIND

greenlight
International Hazard
★★★★★

posted on 8-10-2015 at 03:20

That shouldn't make a difference as far as I know.
I ran my cell in 20 hour intervals with a 4 hour rest every day and had no problems.

Posts: 620
Registered: 3-11-2014
Member Is Offline

Mood: Energetic

Only difference was my cell was 900ml and I used MMO anode and Titanium cathode with 5 volt 20 amp power supply.
Maybe yours is taking longer to form chlorate because of lower current through the cell and you have a much larger cell (2.5L if I am correct).

The only use for an atomic bomb is to keep somebody else from using one.
George Wald

PROFILE FIND

ecos
National Hazard
★★★★

posted on 8-10-2015 at 06:24

I stopped the circuit now. it looks really ugly and very yellow.
I noticed a lot of yellow particles in the bottom of the cell. I identified they are metal because they could be attracted to magnet ! , seems there is erosion to my electrodes .

Posts: 464
Registered: 6-3-2014
Member Is Offline

Mood: Learning !

why the titanium cathode goes dark like this ?

No participation at all for any chlorate ! it is very weird and I am going crazy !

PROFILE FIND

greenlight
International Hazard
★★★★★

posted on 8-10-2015 at 07:49

That is really weird.
My titanium cathode went dark too but that hasn't affected further runs, I just wash it in dilute HCl before use.

Posts: 620
Registered: 3-11-2014
Member Is Offline

Mood: Energetic

The only use for an atomic bomb is to keep somebody else from using one.
George Wald

PROFILE FIND

specialactivitieSK
Harmless
★

posted on 8-10-2015 at 10:09

It is possible by this process to produce copper perchlorate ?

Posts: 40
Registered: 21-10-2014
Member Is Offline

Mood: No Mood

PROFILE FIND

ecos
National Hazard
posted on 13-10-2015 at 16:17

Posts: 464
Registered: 6-3-2014
Member Is Offline

Mood: Learning !

Finally it works. I got some chlorate and I burned it with some sugar and it glow

The connection from the PC power supply to the electrodes were bad and caused voltage drop on each terminal (around 0.8 v) , I also noticed the resistance of the titanium cathode is so high compared to the MMO anode and this also caused voltage drop which made the voltage in the electrolyte very small (< 3v) I think this was the problem.

I used large steel bolt as cathode and fixed the connections between the power supply and the electrode and I started to notice the KClO3 appearing in the second day.

I found some videos on internet on howto make MnO2 anode for chlorate cells :
https://www.youtube.com/watch?v=nvMVIhBmv7M

They use Titanium as substrate , is it possible to use SS as substrate instead of Ti ?

[Edited on 14-10-2015 by ecos]

PROFILE FIND

greenlight
International Hazard
posted on 23-10-2015 at 04:22

Posts: 620
Registered: 3-11-2014
Member Is Offline

Mood: Energetic

Nice to see you got it worked out Ecos...

I finally got everything set up for a perc cell except for the electrodes.
Will one of these platinum coated titanium anodes work or should I wait and fork out the extra money for a lead dioxide one:

http://www.ebay.com/itm/PLATINIZED-TITANIUM-ANODE-PLATING-AN...

Also, for a cathode, can I use straight titanium electrode or do I have to use stainless steel?

The only use for an atomic bomb is to keep somebody else from using one.
George Wald

PROFILE FIND

hyfalcon
International Hazard
posted on 23-10-2015 at 07:45

Posts: 1003
Registered: 29-3-2012
Member Is Offline

Mood: No Mood

That should do fine greenlight. I wouldn't let any of my electrodes sit in electrolyte without power running through them.

Titanium is actually the better the the two.

[Edited on 23-10-2015 by hyfalcon]

PROFILE FIND

ecos
National Hazard
posted on 23-10-2015 at 10:49

Posts: 464
Registered: 6-3-2014
Member Is Offline

Mood: Learning !

> **Quote:** **Originally posted by greenlight**
>
> Nice to see you got it worked out Ecos...
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> I finally got everything set up for a perc cell except for the electrodes. Will one of these platinum coated titanium anodes work or should I wait and fork out the extra money for a lead dioxide one:
>
> http://www.ebay.com/itm/PLATINIZED-TITANIUM-ANODE-PLATING-AN...
>
> Also, for a cathode, can I use straight titanium electrode or do I have to use stainless steel?

As a cathode it is not a problem to use any kind of electrode. SS or Titanium would give almost the same result.
per my experience, SS has better conductivity and this will reduce the voltage loss on it. It also cheap compared to titanium.

after doing the electrolysis you will find a dark layer on the cathode which has high resistance. You would need to remove it with HCl or sandpaper for the next round. This would mean losing of the cathode weight by time.

below is the picture of my cathode (it was pretty shinning before, but looks ugly after electrolysis). I used sandpaper to clean it.

My advice :
1-use TWO SS bolts on each side of the anode. this will generate gasses on each side of the anode rather than one side.
2-save the titanium and plate it with MnO2 or PbO2 (I prepare a thread for this but still needs time). you would be able to use it as anode for another cell.

Try to report the current and results I am waiting for it

[Edited on 23-10-2015 by ecos]

PROFILE FIND

greenlight
International Hazard
posted on 23-10-2015 at 22:31

Posts: 620
Registered: 3-11-

Thanks guys, I will run it with that Pt coated anode and a titanium cathode and post results and specs when finished.

2014
Member Is Offline

Mood: Energetic

The only use for an atomic bomb is to keep somebody else from using one.
George Wald

PROFILE FIND

greenlight
International Hazard
★★★★★

posted on 28-9-2016 at 09:08

Posts: 620
Registered: 3-11-2014
Member Is Offline

Mood: Energetic

I have some experience with making potassium chlorate via electrolysis now and I am ready to attempt a perchlorate cell.
I have a 5v 20 amp power supply from making chlorate and a 5v 40 amp unused power supply. I think I will use the 20 amp power unit as I am only going to use a 1 litre container for the cell as a test unit.

So just to check before I start the cell up:
1# I charge the cell with saturated sodium chloride solution and begin electrolysis with MMO anode and Ti cathode.
2# After the amount of time running that usually gets me a yield of chlorate for this size cell, I change the anode to the platinized one and begin checking samples of the electrolyte with methylene blue for signs of perchlorate.
3# Try and estimate time to near completion using calculations from the various webpages on chlorate and perc manufacture.
4# Double decomp with potassium chloride solution and then recrystallise and wash product until no sign of chlorate left.

Does this sound right?
All help much appreciated

The only use for an atomic bomb is to keep somebody else from using one.
George Wald

PROFILE FIND

ecos
National Hazard
★★★★

posted on 29-9-2016 at 03:09

Posts: 464
Registered: 6-3-2014
Member Is Offline

Mood: Learning !

Hi Greenlight,

I don't think there is a need to use two electrode, the Pt is enough

you can check this video for deep details : https://www.youtube.com/watch?v=O1aWzrRBMjk

why don't u use two cells together, one for chlorate and the other for perchlorate ?

PROFILE FIND

greenlight
International Hazard
★★★★★

posted on 29-9-2016 at 03:44

Posts: 620
Registered: 3-11-2014
Member Is Offline

Mood: Energetic

Hey ecos,
I didnt mean use two electrodes, I mean use the MMO anode and titanium cathode and then when the sodium chlorate levels get high I will swap the MMO for the Pt coated anode...
Then it will just have Pt anode and titanium cathode.

I will check the video out when I get home tonight

The only use for an atomic bomb is to keep somebody else from using one.
George Wald

PROFILE FIND

ecos
National Hazard
posted on 29-9-2016 at 06:53

Posts: 464
Registered: 6-3-2014
Member Is Offline

Mood: Learning !

I totally understand you.

but why to use MMO then Pt if you can use Pt only by converting from NaCl to NaClO4 directly?

hmm let me guess, is it due to that perchlorate will not form till chlorate reach a certain level which is low enough to destroy your Pt anode?

if yes, you can use Pt to get chlorate with certain level then filter them out and use the chlorate to get perchlorate!

it was hard in my cell to change the anode since I used epoxy to fix it.

you will find some basic things that you didn't mention in your steps : 1) boiling water to get over saturated solution .... blah blah

PROFILE FIND

greenlight
International Hazard
posted on 29-9-2016 at 10:28

Posts: 620
Registered: 3-11-2014
Member Is Offline

Mood: Energetic

I thought the least I could use the platinized anode the better in terms of reducing wear on it.
I have heard high chloride levels are hard on it.
I want to go from NaCl to NaClo3 and finally NaCl04 before double decomposition with KCl.

And yes, I forgot to add some small steps like the boiling to attain a saturated solution but I do do that every time I charge the cell electrolyte for my chlorate cell

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The only use for an atomic bomb is to keep somebody else from using one.
George Wald

PROFILE FIND

yobbo ll
National Hazard
posted on 29-9-2016 at 12:07

Posts: 332
Registered: 28-3-2016
Member Is Offline

Mood: No Mood

It's SMALL chloride levels that are hard on the Pt anode.

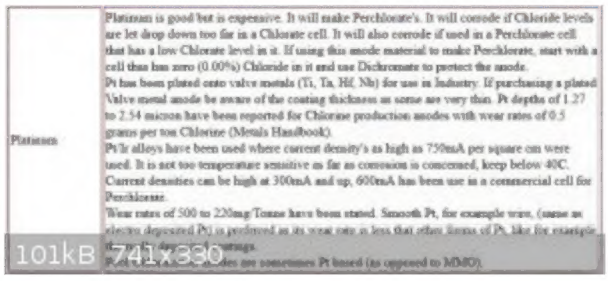
PROFILE FIND

ecos
National Hazard
posted on 29-9-2016 at 23:59

Posts: 464
Registered: 6-3-2014
Member Is Offline

Mood: Learning !

Ref : http://www.oocities.org/capecanaveral/campus/5361/chlorate/a...


Platinum is good but is expensive. It will make Perchlorate's. It will corrode if Chloride levels are let drop down too far in a Chlorate cell. It will also corrode if used in a Perchlorate cell that has a low Chlorate level in it. If using this anode material to make Perchlorate, start with a cell that has zero (0.00%) Chloride in it and use Dichromate to protect the anode.
Pt has been plated onto valve metals (Ti, Ta, Hf, Nb) for use in Industry. If purchasing a plated Valve metal anode be aware of the coating thickness as some are very thin. Pt depths of 1.27 to 2.54 micron have been reported for Chlorine production anodes with wear rates of 0.5 grams per ton Chlorine (Metals Handbook).
Pt/Ir alloys have been used where current density's as high as 750mA per square cm were used. It is not too temperature sensitive as far as corrosion is concerned, keep below 40C. Current densities can be high at 300mA and up, 600mA has been use in a commercial cell for Perchlorate.
Wear rates of 500 to 220mg/Tonne have been stated. Smooth Pt, for example wire, (same as
Platinum
101kB 741x330

I and yaboo are right , for low levels not high levels !

PROFILE FIND

greenlight
International Hazard
★★★★★

posted on 30-9-2016 at 01:28

Posts: 620
Registered: 3-11-2014
Member Is Offline

Mood: Energetic

Whoops, regardless I still want to start with the MMO and then switch over after near conpletion to chlorate.

I am cleaning my titanium cathode in dilute Hcl today and wirebrushing off any buildup of crap on it. The MMO anode was just washed lightly as I don't want to scrape any of the coating off. I am also looking for a larger container for the cell like 2 to 3 litres.

The only use for an atomic bomb is to keep somebody else from using one.
George Wald

PROFILE FIND

ecos
National Hazard
★★★★

posted on 30-9-2016 at 11:34

Posts: 464
Registered: 6-3-2014
Member Is Offline

Mood: Learning !

Good luck and looking forward for your result

PROFILE FIND

greenlight
International Hazard
★★★★★

posted on 28-10-2016 at 08:57

Posts: 620
Registered: 3-11-2014
Member Is Offline

Mood: Energetic

I have finally got the cell finished after a few problems and it has been running for 2.5 hours now.
I am using a 2.25 litre container this time with a 5v 20 amp power supply.
The plan is to run the cell as a sodium chloride cell and convert most of the chloride to chlorate, then recharge the cell solution with sodium chloride and electrolyze again then repeat a couple of times to build up the sodium chlorate concentration. Sodium chloride solubility in water is something like 350g/L @ 25C and sodium chlorate is something around 700g/L @ 25C so once it is converted to chlorate, the cell can hold twice as much dissolved in solution.

Then I will convert to sodium perchlorate with a Pt anode. I can either crystallize the sodium chlorate and start a new cell solution or put the original cell solution straight into the perchlorate cell.

I have calculated the run time at 54% efficiency just to be safe and at my cell size it will take 43.80 hours to convert 86% chloride to chlorate. I will run for 40 hours before recharging with sodium chloride again.

[Edited on 28-10-2016 by greenlight]

The only use for an atomic bomb is to keep somebody else from using one.
George Wald

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